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# Lubrication

A Technical Publication Devoted to  
the Selection and Use of Lubricants

*This Issue*  
★

ANALYSIS OF  
DEPOSITS



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# LUBRICATION

A TECHNICAL PUBLICATION DEVOTED TO THE SELECTION AND USE OF LUBRICANTS

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## ANALYSIS OF DEPOSITS

SEVERAL of the past issues of this publication have briefly considered deposits in lubrication practice as they have related to the particular subject under discussion. The following deals entirely with deposits showing how their various components are separated and identified. It also cites specific cases from several different lubricating systems to illustrate the importance and significance of this type of laboratory investigation.

The nature of deposits is so variable as to defy definition unless one merely states that deposits are undesirable materials found in lubricating systems. In consistency they vary from the semi-liquid sludge type to hard solids depending on their source. For the most part deposit formation is undesirable and those which find their way to the analytical laboratory represent a situation which is or may become a serious lubrication problem. Fortunately for the suppliers of good quality lubricants, these problems are serious in kind but not in quantity. Though this article is wholly devoted to the "bad" cases of deposits, the reader should not surmise that these are commonplace. A major petroleum supplier receives only a relatively small number of such samples from the millions of machines lubricated by his products and the deposit analysis group in his laboratory consists of only a few people.

The purpose of a deposit analysis is to determine the nature and source of its components and the conditions which cause them. There are many factors which cause the formation of deposits. Their origin

may be totally internal or external or a combination of both. They often result from malfunction of equipment and in some cases are the cause of such malfunction. Contamination of the lubricant system and excessively hot or cold operating temperatures cause many deposits. Once the composition of a deposit is known the lubrication engineer can usually locate the source of the difficulty and recommend suitable corrective measures.

Analysis of a deposit and identification of its components involves a different concept than the usual chemical analysis for percentage of an element or functional group. In this type of laboratory investigation the particular physical and chemical forms of the compounds are important in determining what caused the deposit. For example, in addition to determining the amount of iron in a sample it is equally important to know whether its form is metallic (Fe), magnetite ( $\text{Fe}_3\text{O}_4$ ) or rust ( $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ).

### IDENTIFICATION TECHNIQUES

Though deposits vary widely in nature, they are usually separated into one or more of about four major components as shown in Table I. Separating the deposit and identifying its different parts provides a means whereby each component makes a significant contribution in solving the problem. Determining the physical and chemical nature of each component may be relatively simple or very lengthy according to the complexity of the sample.

TABLE I

## Scheme for Separating and Identifying Components of a Deposit

<i>Component</i>	<i>How Separated</i>	<i>Identifying Tests</i>
Volatile Matter	Evaporation in oven at 230°F or water determined by distillation by ASTM D 95-56T.	Unless of special interest this volatile matter is assumed to be water or in some cases light ends of fuel.
Oil	Treat with pentane in small tube, centrifuge, decant and evaporate pentane which leaves oil.	Viscosity, pour, gravity usually done on micro scale. Infrared analysis tells what organic groups are present.
Oxidation Products	Treat pentane insoluble portion from above with mixture of benzene, methyl ethyl ketone and acetone. Centrifuge, decant and evaporate solvents which leaves oxidation products.	Infrared analysis, ash and spectrographic analysis identifies the gum, resin, varnish, metal soap or other complexes present.
Residue	These constituents are the insoluble residue after treating with the above solvents.  The insoluble residue is burned in a combustion train with oxygen and the carbon dioxide absorbed and weighed.	Solvent insoluble residue is examined under the microscope where sand, pieces of metal, fibers and all sorts of extraneous materials are visually identified. X-ray and spectrographic analysis identifies the mineral matter. Amount of carbon dioxide gives total percent carbon both free and that combined with other elements. Ash remaining from the carbon determination gives amount of mineral matter. Per cent ash plus carbon subtracted from 100 shows the per cent loss due to non-metals and volatile metals.

Water is first removed by evaporation (or distillation) and the remaining components separated according to their solubility in various solvents as shown in Table I. After sample and solvent have been well mixed there are a number of ways to separate the insolubles. One can filter thru paper or jena glass crucibles or use a soxhlet extractor. However a more convenient way, especially for small samples, is to centrifuge the mixture at about 3000 RPM in a 12 ml centrifuge tube. This causes the insolubles to separate in the tip of the tube and the solvent layer is poured off into a convenient size micro beaker of from 10 to 25 ml. Evaporation of the solvent leaves the soluble portion for further identification.

Many analytical methods are employed to identify the constituents of a deposit. The amount of a deposit is very often so small that micro-analytical techniques<sup>1</sup> must be employed on samples of a few milligrams. Ordinary physical tests such as gravity and viscosity go a long way toward determining the nature of oils. One of the best ways to characterize or identify oils and other organic materials such as

oxidation products is by use of the infrared spectrophotometer<sup>2</sup>. By absorption (or transmission) of infrared light thru the sample, this instrument gives a spectrogram which shows the *type* of organic groups present. Comparing the spectrogram of the organic material from the deposit with that of known materials makes identification a relatively easy task. Known spectra are usually (1) pure compounds of which thousands of spectrograms are published, (2) commercial substances which have distinctive spectrograms or (3) those accumulated from one's own experience including both unused products and those with a service history similar to that of the sample.

In special cases the mass spectrometer has characterized rubber in a deposit by analysis of the gases given off during depolymerization of the rubber by heat. Specific organic tests are sometimes used to identify particular compounds. An example of this is the use of periodic acid and Schiff's Reagent for detecting ethylene glycol anti-freeze contamination in crankcase oil. The emission spectrograph is very useful in determining metallic elements both qualitatively and quantitatively. It shows the extent to which metals from additives, combustion products,

<sup>1</sup>Magazine Lubrication for January 1957.

<sup>2</sup>Magazine Lubrication for January 1958.

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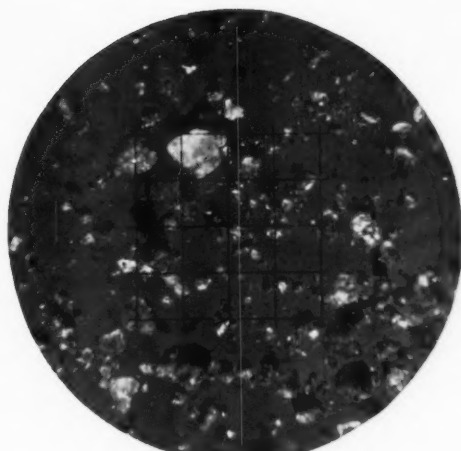


Figure 1 — Photomicrograph of Sand from Vicinity of Overhaul Shops.

wear, corrosion and outside contamination have contributed to the deposit.

In examining deposits or their constituents the stereoscopic microscope with 15 X to 90 X magnifications and the chemical microscope with magnifications up to 1000 X are very useful. For example with the microscope one can observe the presence of sand granules (Figure 1), fibers or metal flakes (Figure 2), also the physical form and size of the constituents which is often of great importance is noted. Metallic iron, for instance, might be in the form of tiny wear particles, filings, turnings or round hardened-steel "shot" (from shot-peening) depending on its origin. Magnetic properties of the deposit are also noted by observing those particles which move in response to a magnet.

X-ray diffraction is one of the most informative tools for analysis of deposits since it gives positive identification of crystalline materials. Knowing the particular chemical formula and crystal structure of a material gives a clue as to the conditions under which the constituent was formed. For example, iron and oxygen combine to form several different oxides according to the particular chemical environment.

### COMPONENT ANALYSIS

#### Volatile Matter

Volatile matter is determined by loss in weight after heating an hour at 230°F. It generally consists of water or fuel dilution or both. The water can be determined either by distillation as in ASTM Method D 95-56T or by Karl Fischer titration. If sufficient sample is available the gasoline dilution may be obtained by ASTM Method D 322-58.

#### Oil

The oil is that component of the deposit which is soluble in a low boiling hydrocarbon such as normal pentane. Since this component usually has undergone the least amount of deterioration, it generally gives the best indication as to what lubricant was employed in the system.

If the problem warrants it, various micro-physical and chemical tests will help to characterize the oil and to determine its degree of deterioration. Gravity, viscosity, ash and a qualitative spectrographic analysis define the type and grade of oil. Neutralization number and infrared analysis indicate the amount of oxidation that has taken place and whether contamination with a totally foreign material has occurred.

#### Oxidation Products

The oxidation products are those materials insoluble in pentane but soluble in solvents such as benzene, methylethyl ketone, and acetone. This component includes gums, resins, asphaltene and other polymeric and oxidized material. They usually result from the deterioration of fuels and lubricants by heat, oxidation and the catalytic effect of metal environment. This component may also contain additive materials that have deteriorated, soaps of iron, copper, zinc and lead or other metallo-organic compounds. Infrared analysis is very useful in characterizing this fraction. Ash determination indicates the relative amount of metallo-organic material present and a spectrographic analysis tells what metals are present. The latter helps to determine whether the oxidation products came from deteriorated additive material in the oil or fuel, corrosion of metal parts, or possibly from contamination with an extraneous material such as a grease.



Figure 2 — Sediment from a Transmission.

- A. Shiny non-magnetic metal chips.
- B. Dull grey magnetic wear particles.
- C. Wood splinter.
- D. Metal sliver.

TABLE II  
Engine Deposits

Sample Source, Engine Place	A Diesel Piston, ring groove	B Diesel Lube oil filter	C Automotive Lube oil screen	D Aircraft Supercharger
Appearance	Oily, black, carbonaceous	Oily, black, thick sludge	Oily, thick, brown granular	Black, carbonaceous flaky solids
Ash, %	16.1	1.1	12.8	22.1
Spectrographic Anal.				
Major	Additive metals	Additive metals	Additive metals	Lead
Minor	—	Potassium, iron chromium, sodium	Iron	Iron, silicon, magnesium, aluminum, phosphorus
Composition				
Volatile, %	2	0.2**	1	—
Oil, %	12	66	52	28
Oxidation				
Prod, %	17	5	25	39
Residue, %	69	29	22	33
Carbon, %	52	91	15	46
Mineral				
Matter, %	22	4	26	40
Volatile, %*	26	5	59	14
X-Ray Analyses	—	—	Lead bromide and chloride	
Microscopic Exam.	Black carbonaceous with few magnetic particles	Black powder, some fibers and bright metal particles	Gray and brown powder, few fibers and magnetic metal particles	Black, granular carbonaceous solids

\*Metals and combined non-metals by difference.

\*\*Water.

In special cases when identity of the organic portion of the soaps is desired, the oxidation products are decomposed by hydrochloric acid and the organic acids extracted by ether. Evaporation of the ether leaves the acids which are identified by infrared analysis. Quantitative micro determinations for chlorine, sulfur and nitrogen are also helpful in identifying the oxidation products.

#### Carbonaceous and Mineral Matter

This component which is usually termed "residue" is that part of the deposit which is insoluble in the various organic solvents. These materials vary greatly and may consist of fuel soot, fibers, sand, rust, metal, metal salts and very highly polymerized oil and fuel decomposition products.

A microscopic examination of the residue supplies a great deal of information as to the nature of the material and its possible source. Shape and size of fibers or other solids are observed and in many

cases the microscope gives their complete identity. Fuel soot may be differentiated from highly carbonized residues from the oil. One can observe whether the siliceous material is fine dust, which could be relatively harmless, or is in the form of larger "sharp" sand granules which cause abrasive wear and equipment failure. The shape and size of metal particles indicates whether they are the result of wear, filings, turnings or even welding beads. Reaction of solids to a magnet is useful in detecting metallic iron and magnetite. In some deposits which are not homogeneous, the microscope shows up small amounts of sand granules and magnetic particles which might easily be overlooked in other methods of analysis.

X-ray and emission spectrographic analyses are most commonly used to show the mineral components present in the residue. X-ray analysis identifies the various crystalline materials present such as sand, mineral dusts, metals, various types of iron oxides,





Figure 3 — Railway Diesel Engine Piston and Rings.

and mineral residues from oil and fuel additives.

How a gasoline engine has been operated can often be deduced by the type of chemical compounds found in the residue. For example halides indicate low temperature operation possibly with a rich mixture while sulfates or oxysulfates are commonly found in deposits from high temperature operation, possibly with a lean mixture.

If iron oxide is present, the *type* indicates the conditions under which it was formed. Magnetite ( $\text{Fe}_3\text{O}_4$ ) is formed by heat or by rusting in the presence of little oxygen such as would occur under a layer of oil. In the presence of moisture and greater exposure to oxygen, rusting is evidenced by the presence of alpha  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  and gamma  $\text{Fe}_2\text{O}_3$ . However, if alpha  $\text{Fe}_2\text{O}_3$  is found without water then either fretting corrosion has occurred or rust which was formed initially has been changed by subsequent heating at high temperature.

Whether or not a particular deposit has caused engine wear can often be evaluated by its abrasiveness. To determine this characteristic, small slabs of metal of various hardnesses are finished with polishing cloths so that all polishing striations are in one

direction. After removal of all the abrasive polishing material, a small amount of deposit is placed on the metal surface with a drop of oil. This mixture is then rubbed with a stick of hard brazil wax at a 90 degree angle to the striations of the metal surface, then carefully removed by flushing with a solvent. Scratches in the metal surface are an indication of the degree of the abrasiveness of the deposit.

Total carbon in the residue is determined by burning with oxygen in a combustion train at about 1400°F to yield carbon dioxide which is absorbed in a special tube and weighed. Carbon, calculated from the weight of carbon dioxide, may come from: soot, highly carbonized oil or other solvent-insoluble organic matter such as rubber, gasket material, paper or fibers. Reweighing the sample boat after combustion gives the amount of mineral matter. Volatile metals and combined non-metals including hydrogen, nitrogen, sulfur and oxygen are calculated by subtracting the sum of carbon plus mineral matter from one hundred.

### CASE HISTORIES

Several specific cases are presented in the following to show how careful analysis of each deposit indicated the cause and pointed to the solution for a wide variety of deposit problems. The case histories in the following tables are grouped, only for convenience of presentation and not cross-comparison according to the type of systems in which they are found. Generally speaking, each deposit has some characteristics which are different from the others shown in the various tables.

### Engine Deposits

Deposits from an engine's crankcase may contain oil, oxidation products and carbonaceous and mineral matter in various ratios depending on the type of service and the particular portion of the crankcase from which they are taken. Diesel engine deposits are markedly different from those in a gasoline engine. Incomplete combustion of heavier fuels accounts for the fuel soot often found in the diesel engine deposits. Table II gives the analyses of deposits from diesel, automotive, and aircraft engines.

#### Diesel Piston

Sample A' in Table II was a very carbonaceous material taken from the ring grooves of the railway diesel engine piston illustrated in Figure 3. Since the mineral matter originated from the additive metals of the lubricating oil, it was concluded that carbonized oil contributed substantially to the formation of this deposit.

#### Diesel Filter

Under Sample B of Table II is shown the analysis of an oily sludge which covered oil filters.

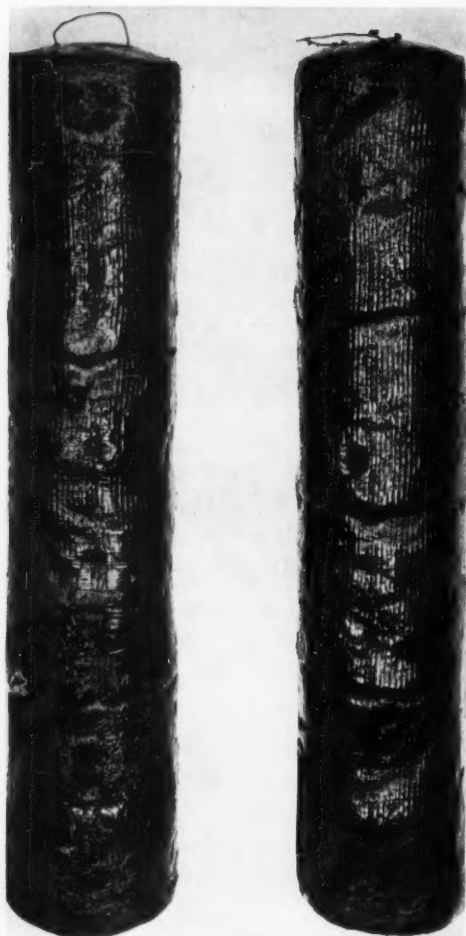


Figure 4 — Oil Filter Cartridges from Railway Diesel Engine.

(Figure 4) from a railway diesel engine. Aside from the oil, the deposit was chiefly fuel soot with only a small amount of water, mineral matter and oxidation products. The presence of sodium and chromium (from a water treating compound) indicated that leakage of cooling water into the crankcase had occurred. A relatively small amount of water in some detergent crankcase oils tends to unite with soot and oxidation products to form a sludge which will markedly reduce filter efficiency.

#### *Oil Pump Screen*

Sample C in Table II presents laboratory data for the thick, brown, oily, granular sludge taken from the automotive oil pump intake screen shown in Figure 5. The material which covered the screen consisted of about 50 per cent oil, 25 per cent oxidation products and 25 per cent of fuel combustion residues. Special solubility data on the oxidation products indicated that they also originated from the gasoline.

Large amounts of fuel degradation products in the crankcase together with information that the oil screen became clogged in a relatively short time led to the conclusion that low temperature operation was chiefly responsible for the deposit. Poor crankcase ventilation resulting from a very dirty breather pipe filter also contributed to this deposit.

#### *Aircraft Supercharger*

After use of an aviation gasoline containing a particular pipeline corrosion inhibitor, it was noted that increased amounts of deposits were forming on the supercharger impeller and housing of large radial reciprocating aircraft engines. In some cases the deposits were sufficiently great to cause reductions in available engine power. Deposits from several engines were analyzed with typical data as shown under Sample D, Table II and in Table III.

TABLE III  
Additional Data on Components of Aircraft Supercharger Deposit

Component	Oil	Oxidation Products	Residue
Percent of Original	28	39	33
Ash, %	6.1	19	40
Spectrographic Analysis			
Major	Lead	Lead	Lead
Minor	—	Phosphorus	Iron, silicon, magnesium, aluminum.
Nitrogen, %	—	2.2	1.0
Separated Organic Acids, %	—	56	21
Infrared Analysis	Carbonyl compounds present.	Separated acids of lead soaps were similar to those in the corrosion inhibitor.	



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TABLE IV

Sample	E	F
Source	Electric motor bearing	Gear and coupling case
Appearance	Gray and white lumpy sludge with some separated oil.	Black resilient solid material
Ash, %	45	4.1
Spectrographic Analysis		
Major	Lead, antimony	Barium, calcium, iron
Minor	Tin, iron, zinc, copper	Sodium, silicon, manganese, phosphorus
Composition		
Oil, %	34	58
Infrared Analysis	—	Oil containing fatty material
Oxidation Products, %	23	12
Appearance	Brown, brittle solid	Pale, clear gum (sticky and stringy)
Infrared Analysis	—	Fatty material
Ash, %	46	—
Spectrographic Analysis		
Major	Lead	—
Minor	Antimony	—
Residue, %	43	30
Carbon, %	12	79
Mineral Matter, %	73	9
Volatile, %*	15	12
Microscopic Examination	Fine gray powder with a few magnetic particles	Small black lumps of resilient solids and several small magnetic metal particles

\*Metals and combined non-metals by difference.

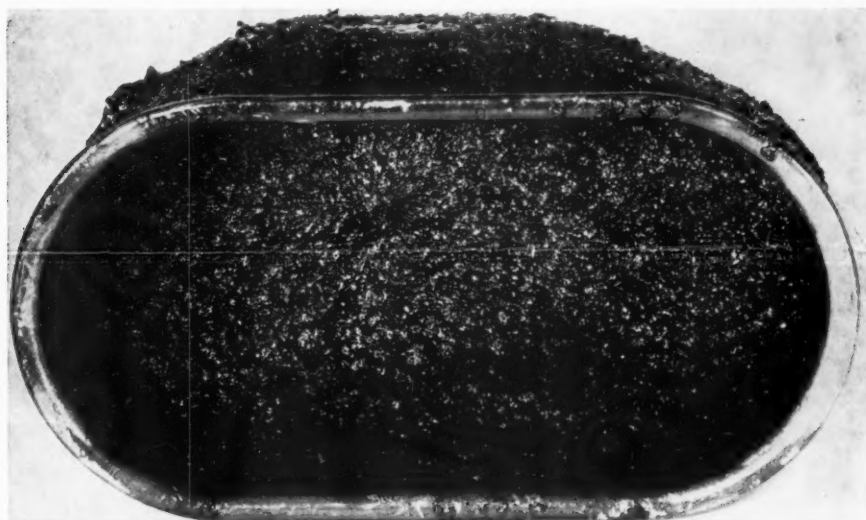


Figure 5—Automotive Oil Pump Screen Plugged with Deposits.

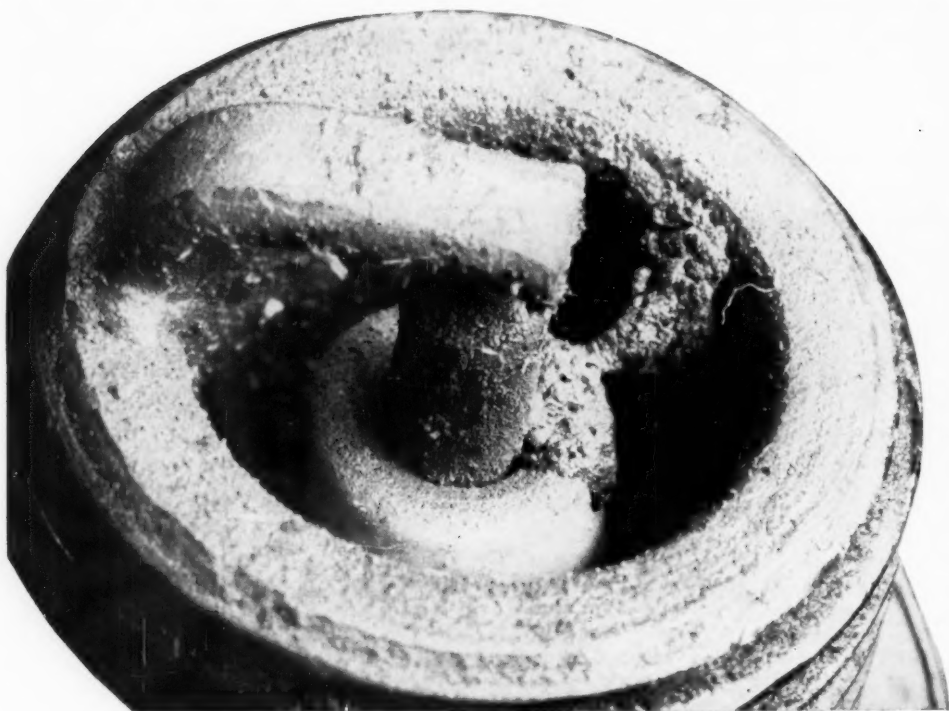


Figure 6 — Spark Plug Shorted by Deposit.

This illustrates the rather extensive laboratory work required for some samples.

The sample consisted of about 50 per cent organic material originating from the gasoline, such as gum, dye and gum inhibitor; approximately 40 per cent lead soaps, and 10 per cent mineral matter (lead compounds other than soap and miscellaneous contamination). The organic acids from the lead soap were similar to the acidic material in the corrosion inhibitor. It then became evident that the inhibitor contained a corrosive constituent which was a critical factor in the formation of the deposits. Subsequent substitution of another type of corrosion inhibitor eliminated this deposit difficulty.

#### *Spark Plug*

Figure 6 shows a spark plug which was "shorted" by bridging of the center electrode to ground with a fused solid. X-ray and spectrographic analyses showed the deposit to be primarily a fusible silicate. This compound has a relatively low melting point and is a good electrical conductor. The silicate resulted from reaction between combustion products and silica dust which had gained entrance to the engine.

### Miscellaneous Deposits

#### *Gear Box*

Sample F of Table IV was a rubbery material found in the gear and coupling cases of a diesel powered motor vessel. It contained a fat, oil and detergent type additive materials, namely barium, calcium and phosphorus compounds. Apparently two or more different lubricants had been placed in the gear system. The deposit resulted from reaction of the fat with a basic detergent additive to form a complex oil-insoluble soap.

#### *Electric Motor*

Analysis of a deposit scraped from a bearing of a 250 horsepower electric motor is shown under Sample E in Table IV. Note that a third of the deposit was oil and that the remaining 66 per cent was oxidation products and mineral residue in the ratio of 1:2. Lead and antimony found in the deposit show that lead-base babbitt bearing metal was damaged and perhaps implicated in its formation.

Relatively large amounts of solvent-soluble oxidation products with a 46 per cent ash indicates that oil degradation products had reacted with some of

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the bearing metal to form metallo-organic compounds termed soaps.

The nature of the deposit suggested two possible causes for its formation which could only be verified by an examination of the bearing and of the used oil in the system. These were:

1. Stray electric currents passing through the bearing would cause pitting and corrosion.
2. These deposits could also have resulted from a hot running motor. Severe oxidation of the oil with the formation of corrosive oxidation products, especially in the presence of a low tin, high lead babbitt bearing would yield this type of deposit.

### *Railway Roller Bearing*

A routine inspection of a few wheel assemblies of railway cars revealed noise and vibrations when the journal bearing assemblies were rotated. Examination of the grease lubricated roller bearings showed a slight film of black deposit on the bearing surface of the rollers which was water soluble.

Data shown in Table V indicated that these small deposits contained approximately 10 per cent "free" glycerine, 20 per cent water-soluble material consisting of colloidal hydrated iron oxide combined with additional amounts of glycerine. There are several references in the literature to the stabilizing effect of glycerine and other polyhydroxy compounds on ferric hydroxide hydrosols. The insoluble residue which comprised about  $\frac{1}{3}$  of the deposit was chiefly iron wear particles and silica.

The following was hypothesized as an explanation for the deposit formation. Water introduced into the grease by condensation or other means promoted rusting of fine metal wear particles. Glycerine, being hygroscopic, probably absorbed some water and became less soluble in the grease. The rust was stabilized by the glycerine from the grease to form colloidal iron hydroxide. These materials were then separated from the grease by centrifugal action. The stabilized iron hydroxide glycerine mixture formed a gummy material which stuck to the bearing rollers, binding the metal wear particles to form the black deposit.

### **Circulating Oil System Deposits**

Though circulating oils are not usually expected to endure the afflictions of high temperatures and combustion products associated with the internal combustion engine, there are several hazards with which they contend. Perhaps the chief of these is the danger of contamination particularly with water. Tables VI and VII present three cases of deposits formed in circulating systems.

#### *Paper Machine*

Sample G of Table VI was taken from the centri-

fuge bowl of the purifying system which maintains clean oil for the moving parts of a large paper making machine. Sample data showed that the material separated by the centrifuge was chiefly solvent-insoluble residue along with water and oil. Cause of this deposit was the water contaminant which led to oil additive separation in this particular service.

#### *Milling Machine*

A black shiny sludge had plugged the strainer of a pump used to circulate soluble oil to a large horizontal milling machine. The sludge had an odor of sewer gas and as shown under Sample H, Table VI, was chiefly an emulsion of oil and water with a small amount of oxidation products and some residue of fibers, dirt, metal particles, and gray film-like material. The filmy material represented only a

TABLE V  
Roller Bearing Deposit

Appearance	Black soft solid.
Benzene Soluble, %	19
Appearance	Pale yellow viscous liquid.
Infrared Analysis	Oil and soaps present.
Alcohol Soluble, %	24
Appearance	Brown and white solids mixed with liquid.
Infrared Analysis	Glycerine and soaps present. Approximately 50 percent glycerine equivalent to 10 percent of the original deposit.
Water Soluble, %	19
Appearance	Dark brown resinous solids.
Alcohol Soluble, %	20
Infrared Analysis	Glycerine and soaps present.
Alcohol Insoluble, %	80
X-ray Analysis	$\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ (Hydrated iron oxide).
Insoluble Residue, %	36
Microscopic Examination	Fine metallic gray magnetic powder with many fibers.
X-ray Analysis	Iron, $\text{SiO}_2$ (Silica).
Loss, %	2

TABLE VI  
Circulating Oil Deposits

Sample System From	G Paper Machine Centrifuge Bowl	H Milling Machine Soluble oil pump strainer
Appearance	Gray and tan chunks like dried shoe polish.	Black shiny sludge
Ash, %	41	12
Spectrographic Analysis		
Major	Additive metals	Sodium, iron
Minor	Bearing metals	Calcium, magnesium, silicon, phosphorus
Composition		
Water, %	19	22
Oil, %	23	59
Oxidation Products, %	9	3
Residue, %	49	16
Carbon, %	16	16
Mineral Matter, %	66	71
Volatile, %	18	13
Microscopic Examination	Chiefly fine tan powder plus few carbonaceous and magnetic particles.	Grayish brown powder, wood fibers, white crystalline lumps of 1000 to 2000 micron in size, also magnetic, dirt and lamellar particles.

\*Metals and combined non-metals by difference.

small portion of the residue and probably was a hard water soap formed from the reaction of the hard water with some of the soluble oil emulsifier. The odor of the sludge indicated that bacteria had caused decomposition in a stagnant pocket in the system which was not readily accessible for cleaning. Plugging of the strainer was probably due to the fibers, dirt and metal particles in the residue.

#### Hydraulic Press

A gummy deposit had accumulated in the hydraulic system of a 300 ton oil-operated press used for moulding plastic parts. A moulding compound used in this manufacturing process was also submitted to determine whether it caused the deposit. Analyses of both materials are shown in Table VII. Eighty per cent of the moulding compound was magnesium silicate, barium sulfate and silica type filler and the remaining twenty per cent was polyisobutylene.

The deposit consisted chiefly of a pale, clear, gummy material together with small amounts of oil, and residue. The latter was mostly calcium carbonate and iron with only a small amount of fillers like those of the moulding compound.

This complaint illustrates the very convenient use of infrared spectroscopy for solving problems since it enables one to identify materials quickly and easily. Figure 7 shows three infrared spectra. The lower two are of components of the deposit, namely oxidation products and oil. The upper spectrum is that of the moulding compound and is essentially the pattern for polyisobutylene. Note that it does not resemble either of the spectra of the components from the deposit. However, the oxidation products spectrum did resemble the spectra of known emulsifiers.

From the infrared examinations and the data shown in Table VII the following conclusions can be drawn:

1. The moulding compound was not implicated in the problem.
2. The hydraulic system oil did not cause the deposit.
3. The deposit contained foreign material similar in nature to emulsifiers or surfactants. This material which possibly originated from the manufacturing process caused the sludging up of the hydraulic press.

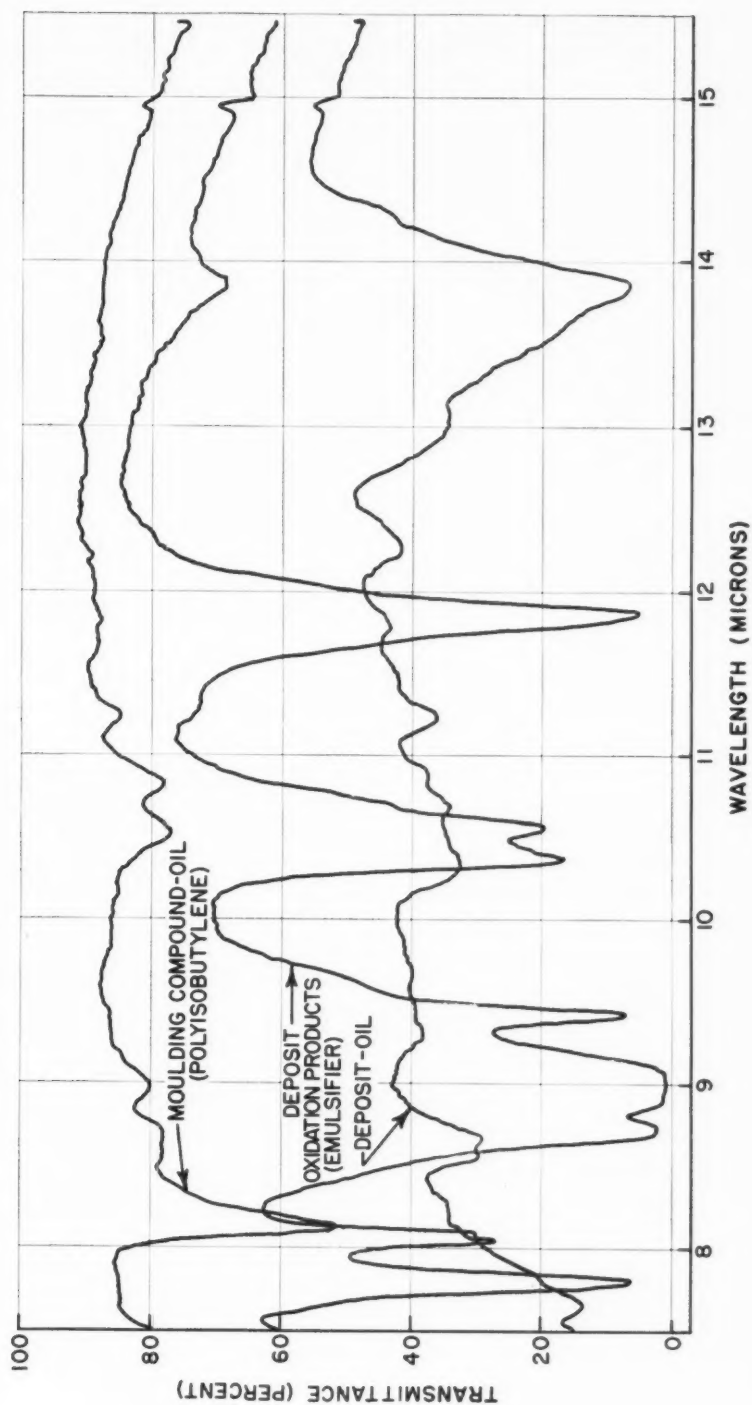


Figure 7 - Three Infrared Spectra.

TABLE VII

	Hydraulic Press Sludge	Moulding Compound
Appearance	Dark brown gummy semi-solid	Soft black pasty material with a few fine fibers.
Ash, %	9.1	70.2
Spectrographic Analysis		
Major	Sodium, calcium, iron	Magnesium
Minor	Magnesium, silicon	Silicon, iron, barium
Composition		
Oil, %	4	23
Infrared Analysis	Hydrocarbons, no polyisobutylene	Polyisobutylene present
Oxidation Products, %	80	—
Appearance	Dark pale clear gum.	—
Ash, %	2.4	—
Spectrographic Analysis		
Major	Sodium, calcium	—
Minor	Magnesium	—
Infrared Analysis	Spectrum similar to that of several emulsifying agents.	—
Residue, %	16	77
Microscopic Examination	Lumps of black solids, many fine asbestos fibers, many magnetic metal particles, a few sand granules and rust particles.	Gray crystalline powder, containing many small, asbestos fibers and a few small magnetic metal particles.
Carbon, %	34	—
Mineral Matter, %	34	—
Volatile, %*	32	—
X-ray Analysis	Calcium carbonate, metallic iron and small amounts of barium sulfate, silica, and magnesium silicate hydrate.	Magnesium silicate hydrate barium sulfate, silica

\* Metals and combined non-metals by difference.

### SUMMARY

Analytical techniques for separating and identifying the components of a deposit have been described. Several deposit case histories have shown the importance of systematic analysis in solving perplexing lubrication problems.

In fairness to those who supply lubricants it should be emphasized again that serious deposits such as those described in this article occur very infrequently. As a matter of fact, petroleum research is continually developing lubricants to meet the demands of ever increasing temperatures and pressures in newly designed machines. Better refined oils

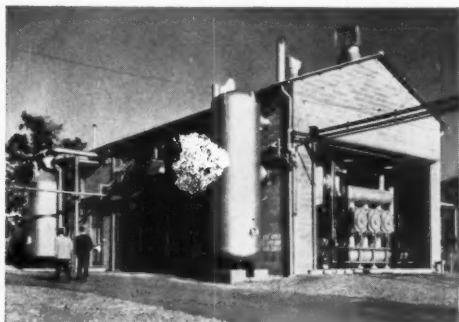
and increased use of additives such as detergents, anti-oxidants and extreme pressure materials help to minimize the formation of deposit materials by reducing wear rates as well as oxidation and settling out of oil insoluble materials.

The analytical chemist not only gathers data but like a detective he wants to know *what, where and why* so that his total information solves the lubrication problem represented by each deposit. As in many of life's complex problems, teamwork is essential and the analyst's success often depends upon the kind and amount of information supplied by the person responsible for lubrication of the machine where the deposit was found.





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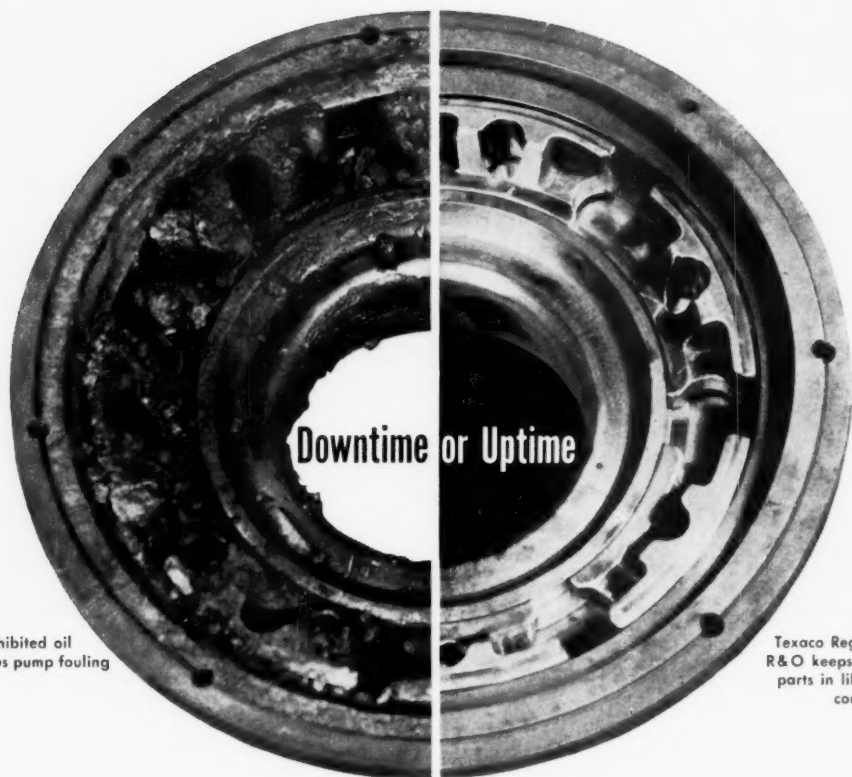
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